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THE TENSILE STRENGTH OF TUNGSTEN, MOLYBDENUM, AND TWO MOLYBDENUM ALLOYS AT VERY HIGH TEMPERATURES

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As part of the general program of research on materials for use at very high temperatures being conducted at the Lewis Research Center of the National Aeronautics and Space Administration, it was found necessary to develop both stress rupture and tensile apparatus for evaluating the properties of these materials at the temperatures of interest. The primary purpose of this discussion is to describe the tensile apparatus that has been developed, and to indicate the properties that have been obtained for sintered tungsten, arc-cast unalloyed molybdenum, and two arc-cast molybdenum-base alloys. The molybdenum-base materials have been evaluated up to a temperature of 3700° F and tungsten up to about 4500° F. A portion of other research on tungsten at the Lewis Research Center is also described.

Figure 1 shows the tensile test apparatus. Because of our interest in brittle materials such as ceramics as well as the more ductile refractory metals, several specifications were placed on the design to permit evaluation of brittle materials. Since some of the experimental brittle materials are difficult to fabricate in long lengths, relatively short test specimens were required. However, in order to minimize grip distortion and welding of specimens to grips, it was desired that the specimen be long enought that it could be gripped outside the hot zone of the furnace. As shown in the central portion of figure 1, these requirements were compromised by use of a five-inch long specimen having only its central portion surrounded by the heater. As will be described later, the heater is an inductively-heated tantalum tube concentric with the test specimen. Since brittle materials are very sensitive to bending stresses resulting from misalignment, special axial loading fixtures were provided. Previous studies at this laboratory have shown that axial loading fixtures of the type shown in figure 1 are more effective than the usual spherical bearings in minimizing eccentric loading. With these loading fixtures, the load is applied through two balls accurately positioned in line with the axes of the loading rods. The test specimens have buttonheads which are fastened to the loading rods by precision holders. The entire assembly is enclosed in a water-cooled vacuum chamber. Lubricated O-rings are used at the top and bottom to seal between the pull rods and the vacuum chamber.

The heater assembly is shown in figure 2. The susceptor is a $1\frac{1}{2}$-inch long seamless tantalum tube that is inductively heated by a water-cooled induction coil. A high-purity recrystallized-alumina tube is cemented to the innersurface of the induction coil to provide both electrical and thermal insulation between the tantalum heater and the induction coil. A 0.010-inch thick molybdenum radiation shield is placed between and concentric with the tantalum and alumina tubes to minimize radiation losses from the heater. This shield contains a longitudinal gap to reduce power loss due to inductive heating of the shield. Induction heating of a susceptor was used in preference to direct heating of the specimen because some of the ceramic materials to be evaluated are not conductive and therefore could not be directly heated.

With this arrangement of test specimen and heater, there is a large temperature gradient along the longitudinal axis of the specimen. For example, at 3600° F the temperature gradient along the central inch of the specimen is about 150° F. The central 1/4-inch of the specimen is fairly uniform, having a gradient of about 20° F at a test temperature of 3600° F. Specimen temperature is measured using a tungsten-molybdenum thermocouple spot welded to the surface of the specimen at its midpoint. The specimen is held for fifteen minutes at the test temperature, and is then loaded to fracture at a crosshead speed of about 1/16-inch per minute. All tests reported herein were conducted in a vacuum of less than 0.5 micron.

The materials evaluated in this investigation were wrought bars of commercially-pure sintered tungsten, arc-cast unalloyed molybdenum, and two arc-cast molybdenum alloys. Information on the chemical composition of these materials is given in the following table:

Material	Chemical Composition (percent by weight)							
	Ti	Zr	Mo	Fe	C	O	N	H
Tungsten	----	-----	0.018	0.021	0.0004	0.0025	0.0015	0.0001
Unalloyed molybdenum[a]	----	-----	Bal.	-----	0.036	------	------	------
Molybdenum-titanium alloy[a]	0.47	-----	Bal.	-----	0.026	------	------	------
Molybdenum-titanium-zirconium alloy[a]	0.46	0.074	Bal.	-----	0.017	------	------	------

[a]Nominal compositions supplied by producer.

Tensile specimens with a 1/4-inch gage diameter were machined from as-swaged or rolled bars and evaluated in the as-received condition. In addition, a few specimens of all materials except the molybdenum-titanium-zirconium alloy were evaluated following a thirty-minute recrystallization anneal at 3800° F in a vacuum of approximately 0.1 micron. The results of the high temperature tensile tests on as-worked specimens of all materials and on recrystallized tungsten are shown in figure 3. Since the molybdenum-base materials recrystallized during the test, even at the lowest test temperature (2500° F), the results of tests on recrystallized specimens were very similar to those for as-worked specimens and are not shown. The curves for the molybdenum-base materials show that the strength advantage of the allows over unalloyed molybdenum decreases rapidly with increasing temperature. Literature data indicate that, at 1600° F, the arc-cast molybdenum-0.45 percent titanium alloy has a tensile strength of 86, 900 psi compared to 52,000 psi for unalloyed molybdenum. At 2500° F this investigation shows that the strength advantage of the titanium alloy has dropped to about 7200 psi (22,500 psi for the alloy compared to 15,300 psi for unalloyed molybdenum. At 3600° F, unalloyed molybdenum and the 0.5 percent titanium alloy have about the same tensile strength, approximately 2200 psi.

Data for commerically pure, sintered tungsten in both the as-swaged and recrystallized conditions are also shown in figure 3. In both conditions unalloyed tungsten is appreciably stronger than even the best available molybdenum alloys over the entire temperature range investigated. At 2500° F tungsten evaluated in the as-swaged condition has a strength of about 49,000 psi compared to 32,000 psi for the recrystallized material. The strength of as-swaged material depends, of course, on the amount of strain hardening retained at the test temperature. While considerable strengthening by cold work is evident at 2500° F, convergence of the strength-temperature curves at about 3100° F indicates that the effects of cold work are annealed out at temperatures above 3100° F. At the highest evaluation temperature, 4470° F, tungsten showed a tensile strength of 4260 psi. In these tests, uncertainty of the temperature measurements is probably less than ±25° F up to 3600° F, but may be as great as ±50° F at temperatures above 4000° F.

Another property of interest is the ductility of these materials at the test temperature. Figure 4 shows the effect of temperature on the reduction of area at fracture of unalloyed tungsten and molybdenum. Data for the materials tested in both the as-worked condition and the fully recrystallized condition are included. In the case of molybdenum, the reduction of area is about 99 percent over the entire temperature range from 2500° to 3700° F. Above the curve are photographs of broken specimens evaluated at the temperature extremes. It will be noted that molybdenum exhibits a very ductile type fracture both at 2500° F and at 3700° F. In tests at all temperatures in this range, localized necking down preceded fracture of the molybdenum-base materials.

Tungsten, however, behaves quite differently. At 2500° F tungsten fractures much like molybdenum, with localized necking and a reduction of area at fracture of about 95 percent for both as-swaged and recrystallized material. However, as temperature increases, the reduction of area at fracture for tungsten decreases reaching an apparent minimum of about 25 percent at 3600° F. At higher temperatures, there is a slight recovery of ductility, the reduction of area being 35 percent at 4470° F. The change in appearance of the fracture zone with increasing temperature is shown by the photographs of broken specimens in figure 4. In the temperature range 3200° F to about 4500° F, the material does not neck, and the fracture appears brittle. However, measurements of elongation and reduction of area show that the material exhibits substantial ductility prior to fracture. Examination of the microstructures of fractured specimens indicates that the change from ductile to brittle-type fracture appears to be associated with a change from predominantly transgranular fracture at low temperatures to grain boundary fracture at the higher test temperatures.

In order to determine if the brittle-type fracture might be characteristic only of the particular material evaluated, commercially pure tungsten from four other producers was evaluated. It was believed that such an evaluation might indicate effects of chemical composition and fabrication history on high temperature ductility. Material from all sources exhibited the same brittle-type failure at temperatures above about 3250° F.

This completes the discussion of data obtained with the high temperature tensile apparatus. Some of the other work we are doing with tungsten will be briefly indicated. It has long been known that recrystallized tungsten is very brittle at room temperature. The ductile to brittle transition temperature of commerically pure tungsten is usually about 500° C. We are interested in determining whether an increase in purity will substantially increase the low temperature ductility of tungsten, and are attempting to procure high-purity material. Electron beam melted tungsten is of particular interest for this purpose, but none has been available to us to date. We are therefore planning the construction of a laboratory-size electron gun melting furnace, and now have in operation a simple zone refining apparatus. Using this equipment, 1/8-inch diameter tungsten rods have been zone melted. These rods, which appear to be single crystals, can be readily bent at room temperature. The shape shown in figure 5 was made by simply bending and squeezing in a vise. Prior to zone melting, the polycrystalline rods were brittle as glass and could not be bent at all at room temperature. We do not yet know whether zone-refined tungsten will be ductile when polycrystalline.

In addition to the investigation of the influence of purity on the ductility of tungsten, a study of the alloying of tungsten has been initiated. Its prime objective is to achieve tungsten-base materials with increased strength at 3000° F and above. Alloys with improved ductility and oxidation resistance at lower temperatures are also being sought. To date a few buttons of tungsten alloys have been nonconsumably arc-melted and attempts have been made to hot roll the buttons. These attempts have been largely unsuccessful. Higher working temperatures appear desirable and equipment is being designed to permit hot rolling at temperatures up to about 4000° F.

It is planned to use consumable electrode melting for most of the tungsten alloy programs. To data we have been trying to melt pure tungsten in the consumable electrode furnace. Several ingots have been produced that are approximately $1\frac{1}{2}$ inches diameter by 3 to 5 inches long. One of these ingots is shown in figure 6. This ingot was melted in vacuum at a pressure of approximately 25 microns using direct current. The water-cooled copper mold was lined with 5 mil tungsten sheet in order to reduce the rate of heat extraction from the mold and maintain a molten pool. The figure shows a transverse section very near the bottom of the ingot. Except at its periphery the ingot appeared sound, containing only a very small amount of porosity. The as-cast hardness was about Rockwell C-28. Attempts to extrude our ingots and an ingot from a commerical source are now being conducted at two outside laboratories.

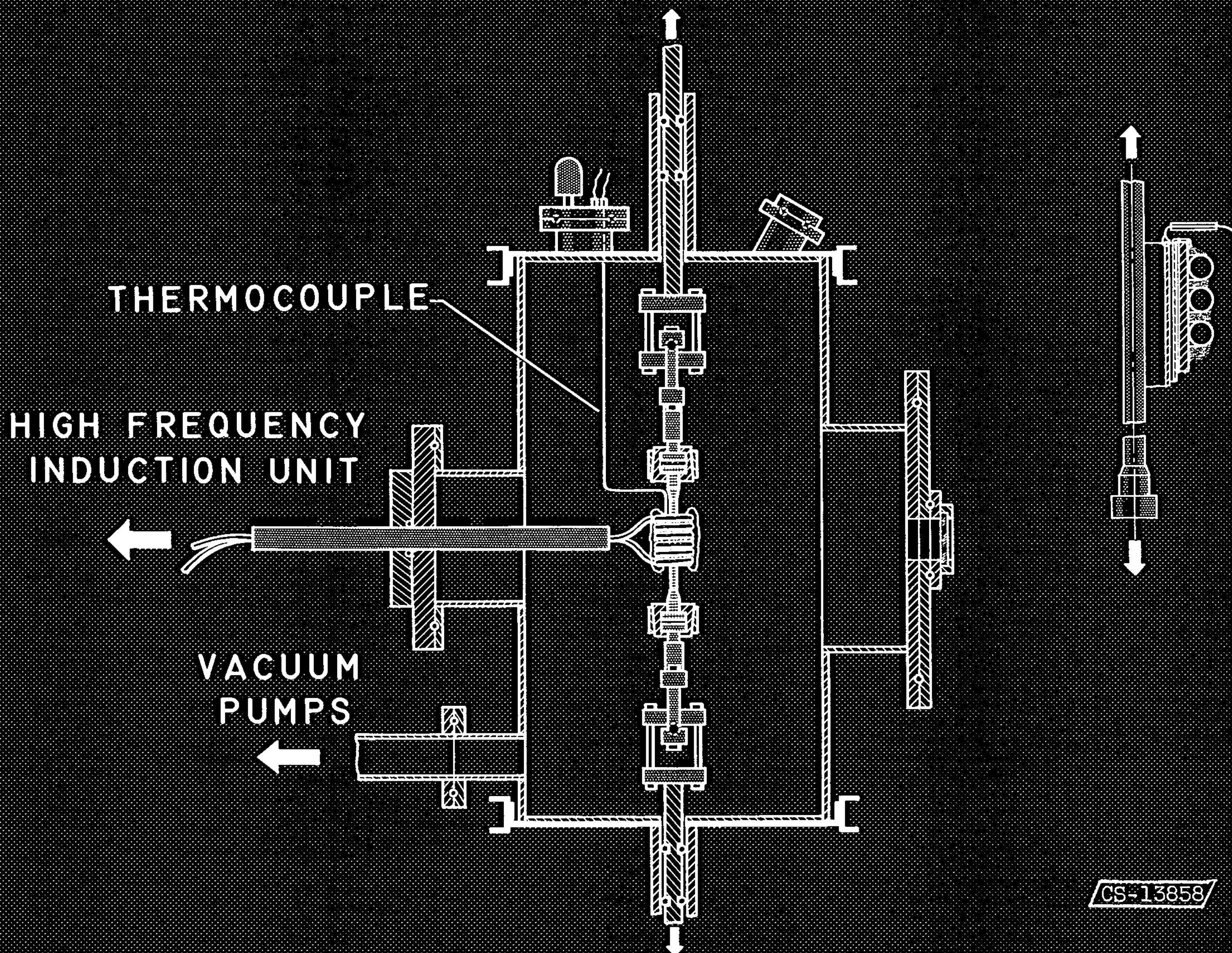

Figure 1. - High temperature tensile test apparatus.

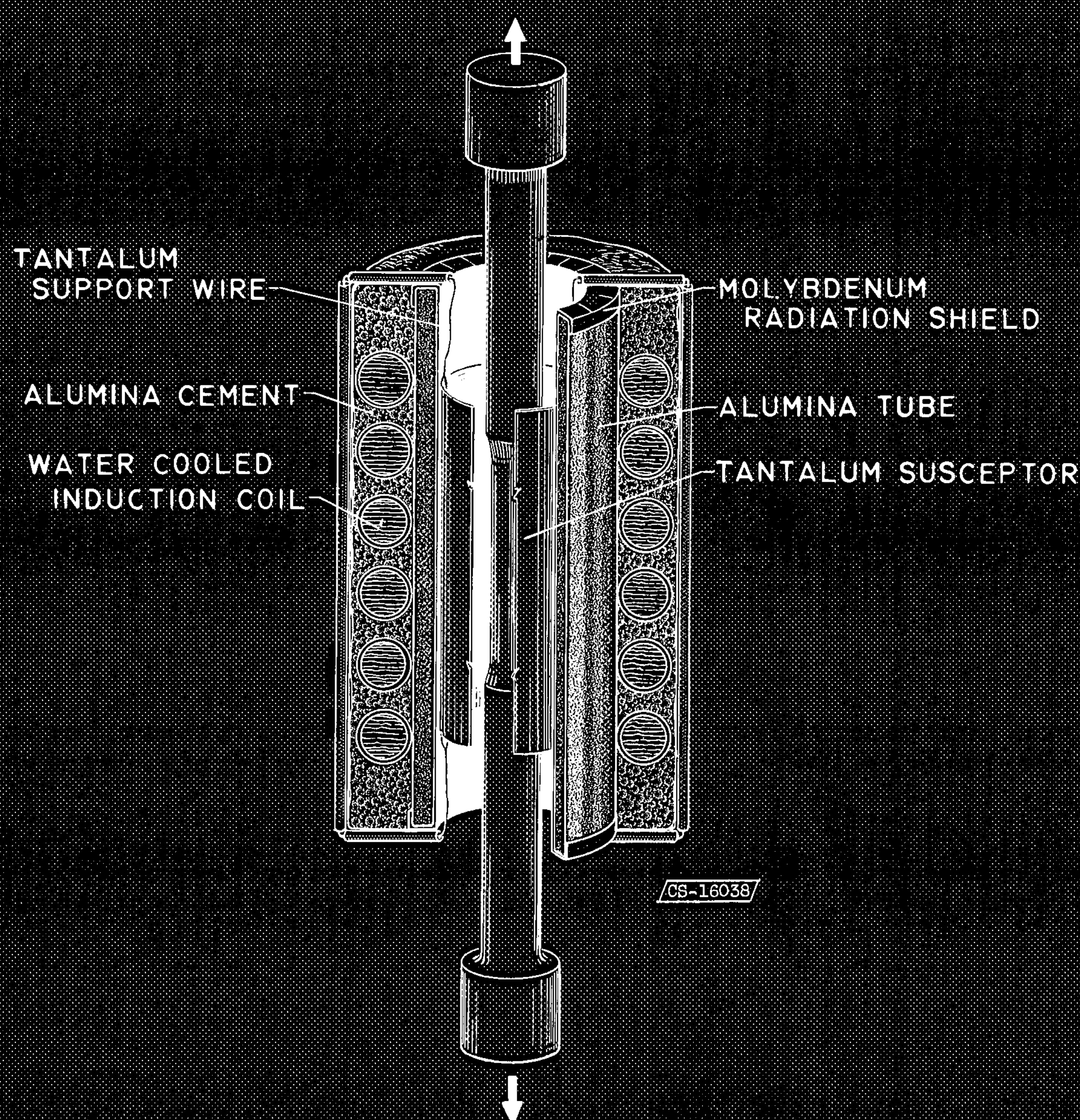

Figure 2. - Heater assembly.

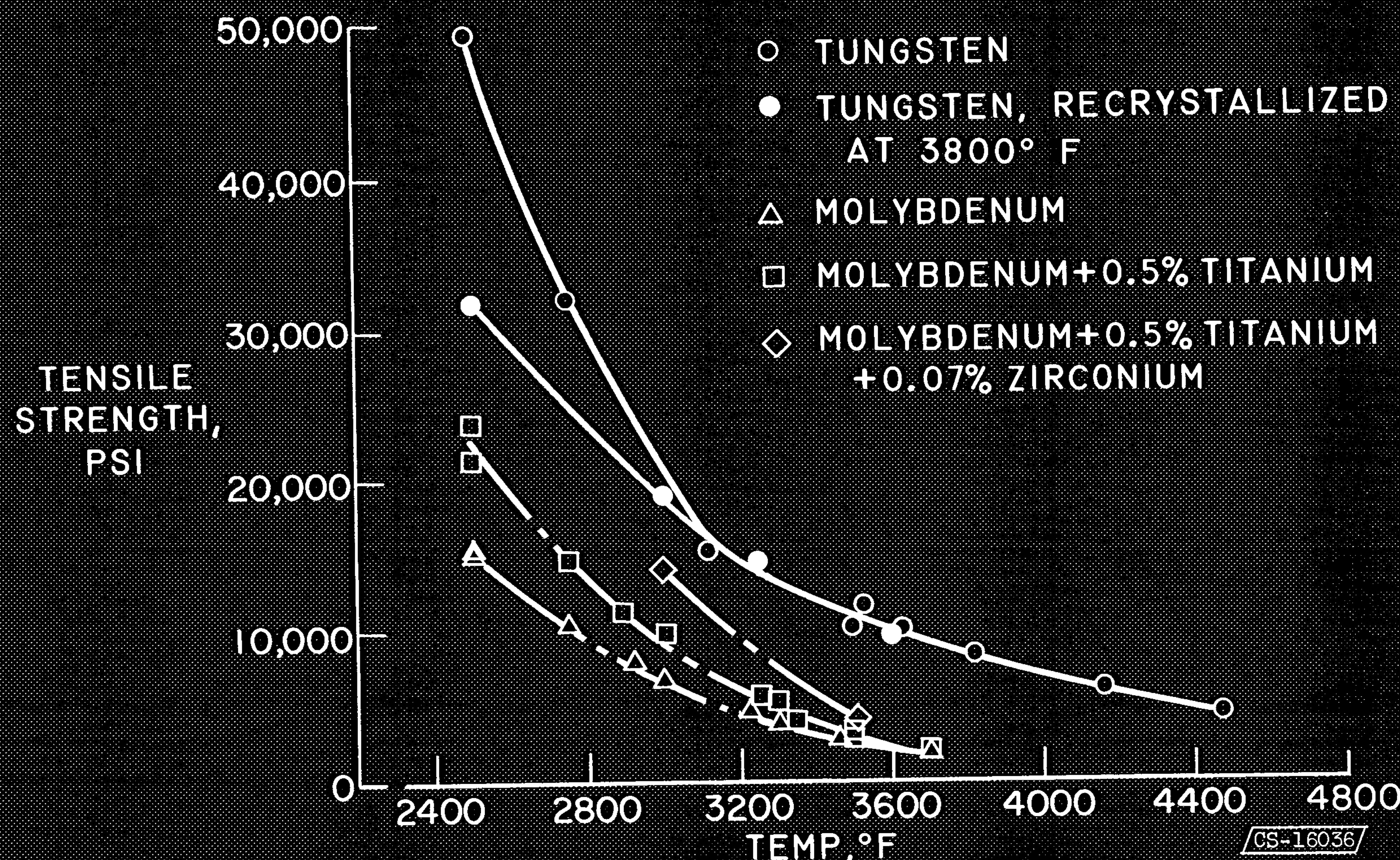

Figure 3. - Tensile strength of tungsten, molybdenum and molybdenum alloys at high temperatures.

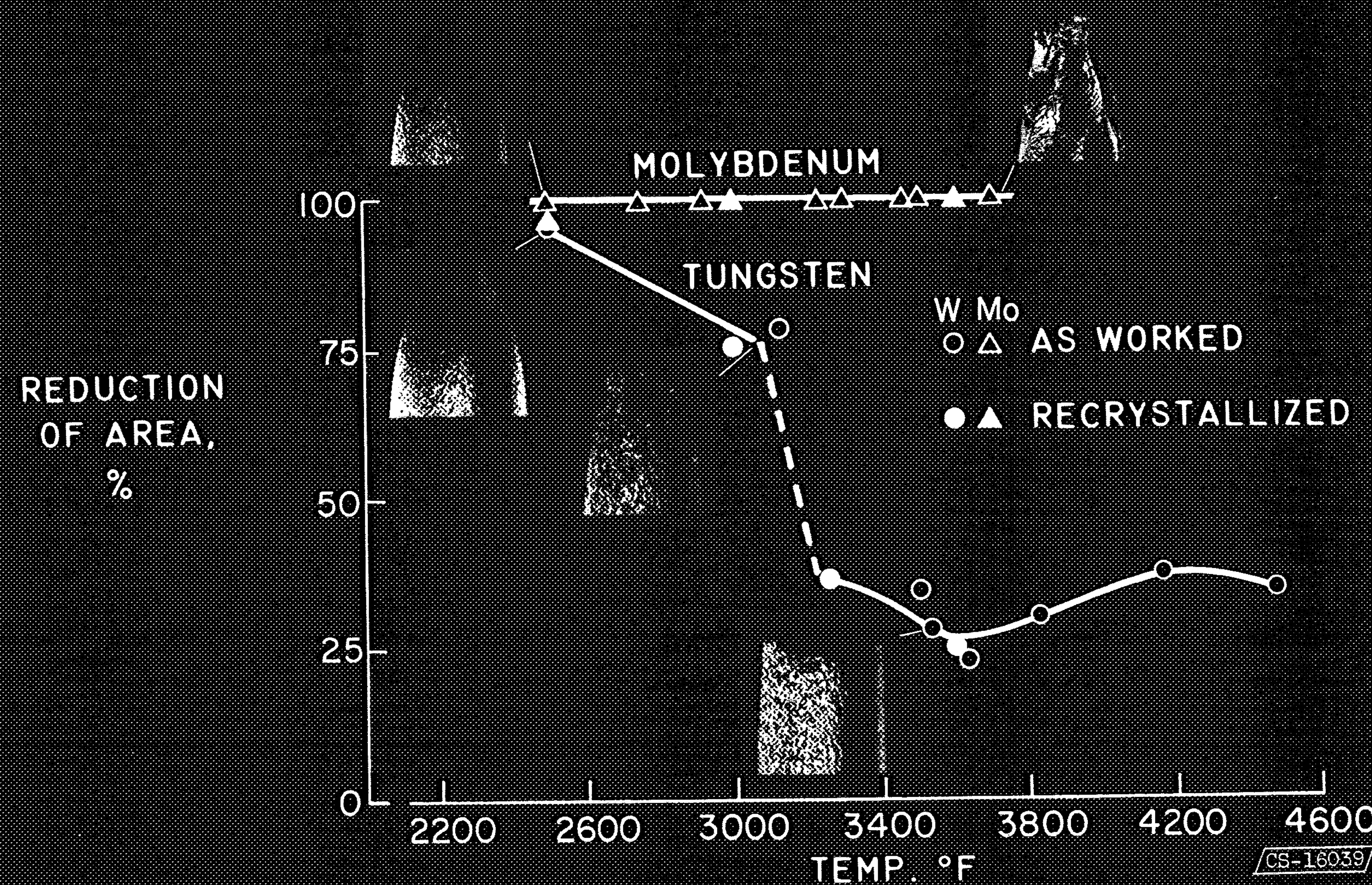

Figure 4. - Effect of temperature on reduction of area of tungsten and molybdenum.

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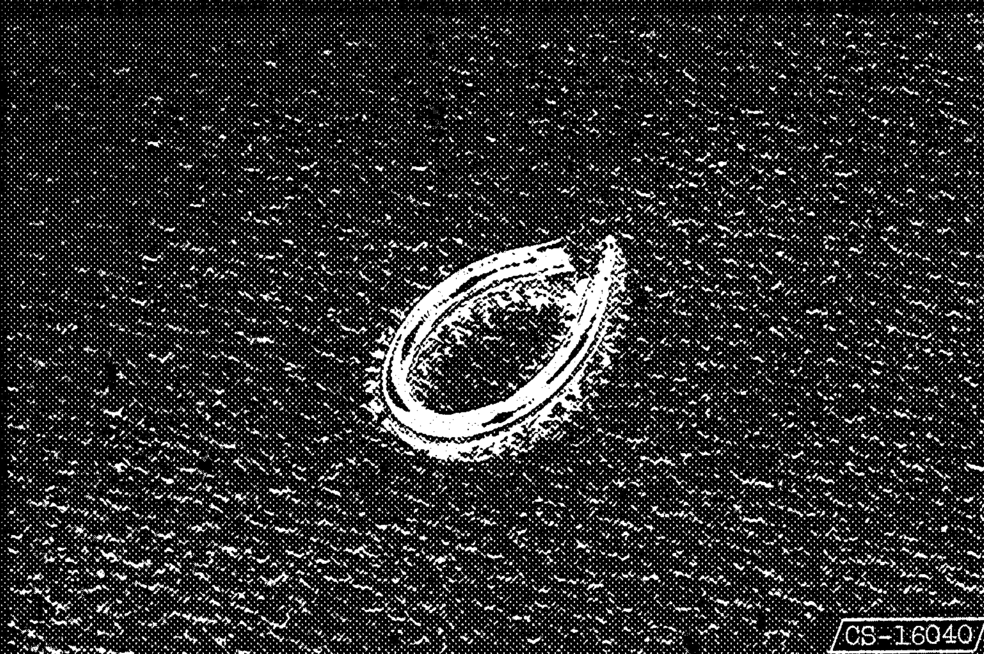

Figure 5. - Ductility of zone refined tungsten.

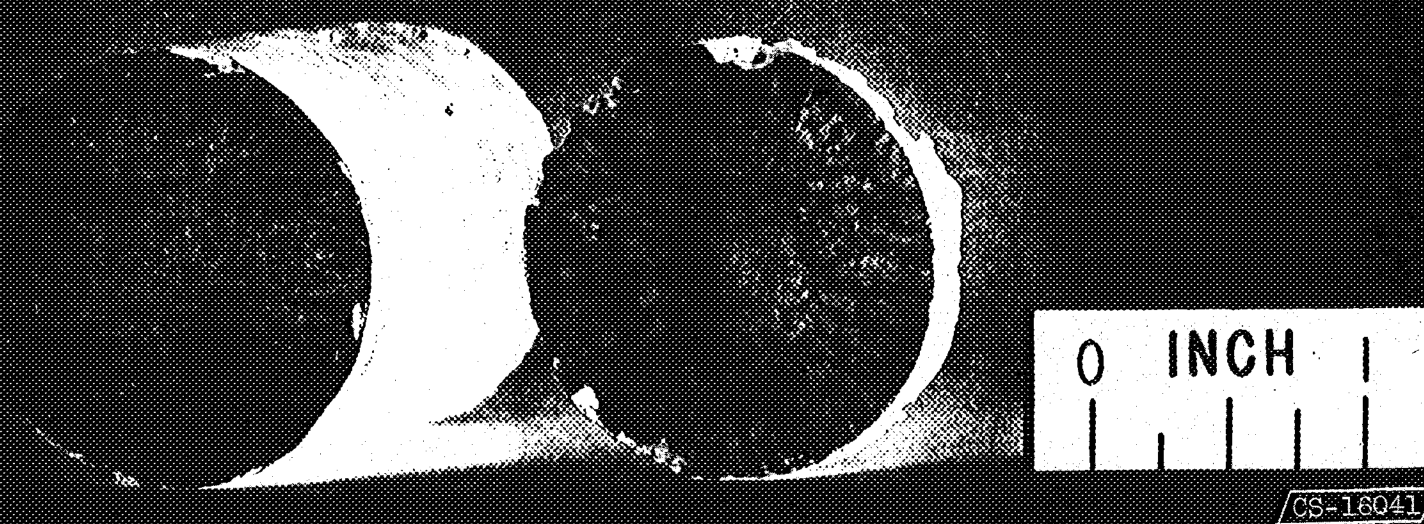

Figure 6. - Grain structure of 1-1/2-inch diameter vacuum arc-cast tungsten ingot.

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